METHOD FOR MAKING FUSED CERAMIC ARTICLES OF NEAR NET SHAPE

FIELD OF THE INVENTION

The present invention pertains to the field of binders used to form fused ceramic articles.

BACKGROUND

Many processes for forming fused ceramic bodies begin by forming porous green bodies, which are composed of a batch material that includes particulates of inorganic compounds held together with binders. One of the final steps in these processes is a firing step in which the original particulates are reacted to become a fused ceramic body. Often all or most of the binders burn out in this firing step. These processes have constraints, including green bodies with large particulates and large pores (e.g., greater than 10 microns average size), that experience high sag in firing due to a low ratio of sintering forces relative to gravity forces. Green bodies that have high porosity can have very low strength in firing and will often fracture or crumble before particles fuse.

One approach taken to address the above constraints is adding one or more high temperature binders to the material mixture of the body prior to forming the body. An organometallic pre-ceramic precursor binder can be added to the batch material, which is formed into the green pre-formed bodies. The ceramic precursor binders contribute to the overall ceramic content of the finished part. In another approach, a pre-formed green body can be heated to burn out traditional binder and then infiltrated with a pre-ceramic organic or inorganic binder material that can be a monomer, oligomer or polymer in a solvent. This binder can include an organometallic material.

Sol gel materials have been made, for example, using metal alkoxides or metal salts, organic solvents and acid or base catalysts. While much interest in these materials was generated when they were first developed decades ago, glass or crystalline bodies formed using sol gel materials were expensive and difficult to process. For example, the drying step used to prepare monolithic fused glass bodies was carried out by very slow drying or expensive supercritical drying. As a result of the difficulties of using sol gels to form large monolithic bodies, sol gel technology has been applied to areas including thin films and fiber coating. For example, ceramic matrix composites can be made by infiltrating fiber performs with ceramic sols to form a ceramic matrix material.

SUMMARY

The present invention uses an impregnation and drying treatment that adds high temperature binder material to an already formed porous green body or pre-form. The green body is composed of a particulate batch material. The batch material includes inorganic compounds that are capable of forming ceramic crystalline or glass phases, and binder material. The pre-formed green body used in the inventive method has a shape that is retained when the body is later fired and transformed into a fused ceramic (i.e., the inventive method produces fused ceramic articles of near net shape). In general, the inventive treatment is characterized by two steps: 1) the formed porous green body is infiltrated with a homogeneous sol gel precursor solution to fill the pores of the green body with the solution, and 2) the infiltrated body is dried to concentrate the solution and then polymerize the concentrated solution. The treatment can then proceed to firing. Alternatively, the impregnation and drying can be repeated one or more times before firing.

The sol-gel precursor infiltrating solution is a mixture of monomers and oligomers formed by reacting pre-ceramic organometallic compounds or pre-ceramic metal salts and organic solvent in the presence of an acid catalyst. The porous green bodies can be infiltrated by being submerged in the infiltrating solution. The infiltrated bodies are removed from the solution and dried to remove the solvent and then to form a polymer of the pre-ceramic compound in the infiltrated bodies. The dried bodies are fired at a temperature to form fused ceramic articles, for example, substrates for catalytic converters, flow mixers for fluid streams, and filters for filtration of liquids.

One embodiment of the present invention features providing shaped porous green bodies of particulate batch material. The batch material includes inorganic source compounds and binder. An infiltrating solution which is a sol gel precursor mixture of monomers and oligomers, is formed by reacting pre-ceramic organometallic compounds or pre-ceramic metal salts and organic solvent in the presence of an acid catalyst. The porous green bodies are infiltrated with the infiltrating solution to fill the pores of the green bodies with the infiltrating solution. The infiltrated bodies are dried to remove most of the solvent and then to form a polymer of the pre-ceramic compound in the bodies. The dried bodies are fired at a temperature to form fused ceramic articles that include first ceramic material derived from the inorganic source compounds fused with second ceramic material derived from the pre-ceramic compound.

Referring now to more specific features of the inventive method, the infiltrating solution employs the pre-ceramic compound in an amount effective to enable the second ceramic material to comprise 2-20%, in particular 5-10% by weight, of each ceramic article. The pores can be filled with the infiltrating solution without applying pressure by submerging the porous bodies in the infiltrating solution for not more than 1 minute. The infiltrating (e.g., submerging) step is conducted on the room temperature porous green bodies that have not been heated above 150° C. During the drying step the polymer can be formed in necking regions between contiguous particles and in small pores of the infiltrated bodies. The submerging and drying steps can be carried out at least two times, which may further reduce sagging and shrinkage of the fired bodies.

As to the specific aspects of the composition of the sol gel precursor solution, the organometallic compounds may include, but are not limited to, at least one metal alkoxide such as, but not limited to, magnesium alkoxide, aluminum alkoxide, zirconium alkoxide, phosphorous alkoxide, titanium alkoxide, niobium alkoxide, tantalum alkoxide, and the like. Alternatively, the organometallic compounds may include boron alkoxide, silicon alkoxide, and/or phosphorous alkoxide. The metal salts include, but are not limited to, at least one of magnesium nitrate, lithium nitrate, calcium nitrate hydrate, yttrium nitrate hydrate, and the like. The solvent includes at least one of 2-methoxyethanol, ethanol and n-propyl alcohol. The acid catalyst is an aqueous nitric acid solution containing 60-85 weight % nitric acid.

The inventive method results in characteristic properties. Most notably, strength of the fused articles is maintained while reducing distortion during sintering/firing as defined below. The impregnation and drying treatments can be effective in both improving fused body strength and in reducing sag that develops during sintering/firing. The treatment is simple and fast, and creates fused ceramic bodies of near-net-